International Journal of Advanced Engineering Research and Science (IJAERS)
Peer-Reviewed Journal
ISSN: 2349-6495(P) | 2456-1908(O)
Vol-9, Issue-8; Aug, 2022
Journal Home Page Available: https://ijaers.com/
Article DOI: https://dx.doi.org/10.22161/ijaers.98.53

Innovative cocktails: Physical-chemical profile during storage

Coquetéis inovadores: Perfil físico-químico durante o armazenamento

Fábio Pereira de Souza[1], Mariana Teixeira da Costa Machado[2], Alexandre Santos de Souza[3], Sandra Regina Gregório[4], Davy William Hidalgo Chavez[5], Lara Bruna Brito Castro de Souza[6], Karina Costa[7], Luiz Fernando Oliveira Maia[8], Jairo Lisboa Rodrigues[9]

[1]Laboratory Technician of the IFNMG/Campus Salinas 39560-000 Salinas, MG- Brasil.
[2]Professor at UFRRJ/Campus Seropédica, 23890-000 Seropédica, RJ- Brasil.
[3]Professor at IFNMG/Campus Salinas 39560-000 Salinas, MG- Brasil.
[4]Professor at UFRRJ/Campus Seropédica, 23890-000 Seropédica, RJ- Brasil.
[5]Professor at UFRRJ/Campus Seropédica, 23890-000 Seropédica, RJ- Brasil.
[6]Laboratory Technician of the IFNMG/Campus Salinas 39560-000 Salinas, MG- Brasil.
[7]Laboratory Technician of the IFNMG/Campus Salinas 39560-000 Salinas, MG- Brasil.
[8]Professor at IFNMG/Campus Salinas 39560-000 Salinas, MG- Brasil.
[9]Professor at UFVJM/Instituto de Ciência, Engenharia e Tecnologia/Campus 39803371 - Teófilo Otoni, MG - Brasil

Received: 14 Jul 2022,

Received in revised form: 03 Aug 2022,

Accepted: 09 Aug 2022,

Available online: 31 Aug 2022

***Keywords*— grain alcohol, physicochemical analysis, mixed alcoholic beverages, cachaça, shelf life.**

***Palavras-chave*— álcool de cereais, análises físico-químicas, bebidas alcoólicas mistas, cachaça, vida de prateleira.**

***Abstract*— For the improvement and inclusion of new food technologies in the market, several evaluations must be carried out carefully in a new product, from its formulation, physical-chemical and microbiological tests to acceptance by the final consumer. This study aimed to evaluate the formulation of bottled alcoholic beverages in relation to physical-chemical aspects and stability during storage at two temperatures, for a period of up to 150 days, analyzing the physical-chemical parameters. Mixed alcoholic beverages, caipirinha, margherita and dulce de leche flavors, were evaluated regarding mandatory parameters of identity and quality standards of the legislation, quantification of chemical elements (Mg, k, Mn, Fe, Cu, Pb, As, Cd and Sn). For the stability study, the Accelerated Shelf-Life Test (TAVP) was performed, the samples were stored at temperatures of 30 and 40 °C in a BOD-type oven, in the absence of light and evaluated at regular intervals of time in relation to the parameters physical-chemical properties and color, in order to verify if there was a significant difference between the standard sample (beginning of storage) in relation to each elapsed temperature and time. With the exception of the caipirinha, which did not reach the minimum limit of 1g/100 mL of lemon juice, but this parameter can be easily corrected. The drinks remained stable throughout the storage time, the physico-chemical parameters analyzed of the three samples did not show changes in behavior at temperatures of 30 and 40 °C, when compared to each other, despite having presented a significant difference by the Tukey test and the non-*

parametric Kruskal Wallis test at a significance level of 5%. It was noticeable in the visual analysis of color and the comparisons of color parameters (L, Chroma and HUE) that there was a color variation over the storage time, however, this did not influence the shelf life, which was estimated to be greater than 150 days. Thus, it can be concluded that the drinks were stable and commercially acceptable.

Resumo— *Para o aprimoramento e inclusão de novas tecnologias de alimentos no mercado, diversas avaliações devem ser realizadas criteriosamente em um novo produto, desde sua formulação, testes físico-químicos e microbiológicos até a aceitação pelo consumidor final. Este trabalho teve como objetivo avaliar a formulação de bebidas alcoólicas engarrafadas em relação aos aspectos físico-químicos e estabilidade durante o armazenamento em duas temperaturas, por um período de até 150 dias, analisando os parâmetros físico-químicos. As bebidas alcoólicas mistas nos sabores caipirinha, margherita e doce de leite foram avaliadas quanto aos parâmetros obrigatórios de identidade e padrões de qualidade da legislação, quantificação de elementos químicos (Mg, k, Mn, Fe, Cu, Pb, As, Cd e Sn). Para o estudo de estabilidade, realizado o Accelerated Shelf-Life Test (TAVP), as amostras foram armazenadas nas temperaturas de 30 e 40 °C em estufa tipo BOD, na ausência de luz e avaliadas em intervalos regulares de tempo em relação aos parâmetros propriedades físico-químicas e cor, a fim de verificar se houve diferença significativa entre a amostra padrão (início do armazenamento) em relação a cada temperatura e tempo decorridos. Com exceção da caipirinha, que não atingiu o limite mínimo de 1g/100 mL de suco de limão, mas esse parâmetro pode ser facilmente corrigido. As bebidas permaneceram estáveis durante todo o tempo de armazenamento, os parâmetros físico-químicos analisados das três amostras não apresentaram alterações de comportamento nas temperaturas de 30 e 40 °C, quando comparadas entre si, apesar de terem apresentado diferença significativa pelo teste de Tukey e pelo teste não paramétrico de Kruskal Wallis ao nível de significância de 5%. Foi perceptível na análise visual de cor e nas comparações dos parâmetros de cor (L, Croma e HUE) que houve uma variação ao longo do tempo de armazenamento, porém, isso não influenciou na vida útil, que foi estimada em superior a 150 dias. Assim, pode-se concluir que as bebidas foram estáveis e comercialmente aceitáveis.*

I. INTRODUCTION

Alcoholic beverages have been manufactured for hundreds of years and feature in some passages in human history. Although they have been produced by hand for centuries, in Brazil, today they are obtained through industrial processes (Gonzaga & Moreira, 2008). Cachaça or sugar cane spirit, a genuinely Brazilian drink, the main ingredient of caipirinha, a famous Brazilian drink (FONSÊCA, 2020), is the second most consumed drink in the Brazilian market and the third most consumed distillate in the world. The national production of cachaça is 1.5 billion liters/year and has remained constant in recent years, it stands out as one of the most produced and consumed beverages in Brazil, with about 1.5 billion liters per year. (VIANA, 2016). It is estimated that there are 40,000 cachaça producers in Brazil, generating 6,000 direct jobs, with an annual income of US$2 billion (Naconha, 2021), with more than 5,000 registered brands and about 30,000 producers across the country (MIRANDA, 2007).

According to Filho (2003), for a good quality alcoholic beverage, appropriate quality control procedures must be adopted from the beginning of the process, starting with the choice of raw material, passing through the processing, fermentation, distillation and careful storage of the final product. obtained. The identity and quality standards of an alcoholic beverage can be

evaluated considering the physical-chemical, microbiological and sensorial aspects.

Mixed alcoholic beverages or alcoholic cocktails or alcoholic cocktails are drinks existing in the national territory and supervised by the Ministry of Agriculture, Livestock and Supply (MAPA), through the provisions of Decree N° 35 (BRASIL, 2010a), RDC 05(BRASIL, 2013), RDC 45 (ANVISA, 2010), (RDC nº 2, de 15 de janeiro de 2007 and (IN 13, 2005).

According to current Brazilian legislation, a drink made up of fruit, pulp or plant extract is a drink obtained by mixing juices, pulps or plant extracts, together or separately, with a product of animal origin, with a predominance in its composition of a product of vegetable origin, with or without sugars (BRASIL, 2009).

The legislation allows a wide combination of its ingredients, varying between maximum and minimum concentrations, causing less or greater acceptance of consumers between drinks with the same denomination, however, from different producing industries. There are few formulations on the market; these drinks are usually made at home for immediate consumption, by people who enjoy it, at parties by bartenders or made by mixologists. Ready-to-eat commercialized formulations can optimize the entire production chain process in terms of reducing waste generated, preparation time, long lines (when produced by bartenders at parties), increased standardization, among other aspects.

For the development of food products, a series of quality assessments are necessary, including physical-chemical, sensory and microbiological analyses, which can intervene in the different stages of the product's life cycle (Giménez et al., 2012); (Schneider et al., 2018). Products can undergo changes during storage, such as microbiological deterioration, lipolytic oxidation reactions and nutrient degradation, causing the non-acceptance of the product by the consumer (Schneider et al., 2018).

In this context, the objective of the present work is to evaluate three formulations of mixed alcoholic beverages, which will be placed in the market by the company Asscorp Alimentos LTDA, regarding the physicochemical characteristics in relation to the identity and quality standards (IN Nº 13, de 2005) (Decree No. 6,871 of June 4, 2009).

II. MATERIALS AND METHODS

2.1 Materials

Three mixed alcoholic beverages were analyzed in this work: caipirinha, margherita and dulce de milk cocktail. The proposed formulation of margherita has as ingredients demineralized water, cereal distillate, sugar, lemon juice, orange juice, antioxidant and stabilizer. The dulce de leche cocktail in its formulation has demineralized water, cereal distillate, skimmed milk jam, sugar, antioxidant and stabilizer. For the caipirinha, the base was used, which is cachaça, sugar, lemon juice, natural lemon aroma (essential oil), antioxidant and stabilizer. The samples were made at the company Asscorp Alimentos LTDA and kindly provided to carry out the project, Asscorp is located in the city of Salinas, Minas Gerais-Brazil), a partner company in the project.

III. METHODOLOGIES

3.1 Study Design

In the first batch, 72 samples of the three beverages were manufactured and packaged in glass bottles with a volume of 750 mL, being 24 mixed alcoholic drinks of caipirinha, 24 of dulce de milk and 24 of margherita. These 72 samples were transferred to the physical-chemical analysis laboratory of the IFNMG-Campus Salinas and placed in an oven for storage at 30 or 40 ºC, in the absence of light, until the physical-chemical analysis was carried out, where monthly, for 6 months until the final period of 150 days, 2 samples of each of the 3 flavors, at the two storage temperatures, were removed from the ovens intended for physical-chemical analysis.

Initially, the batch samples (time zero) of the three drinks were subjected to mandatory analyzes by the Ministry of Agriculture to verify that the drinks meet the identity and quality standards according to (Brasil, 2019). For the sample of caipirinha, the parameters of alcohol content, in %, in v/v, at 20 ºC, lemon juice, expressed in %, with 5% acidity titratable in citric acid, in g/100 g, sugar in sucrose expressed in g/L, coefficient of congeners (expressed in mg/100mL of anhydrous alcohol), aldehydes, total esters, higher alcohols (expressed as the sum of n-propyl alcohol, isobutyl alcohol and isoamyl alcohol), and furfural (furfural + 5-hydroxymethylfurfural), analysis for organic contaminants: methyl alcohol (methanol) in mg/100 mL of anhydrous alcohol, ethyl carbamate, in µg/L, acrolein (2-propenal), in mg/100 mL of anhydrous alcohol, n-butyl alcohol (1-butanol), in mg/100 ml of anhydrous alcohol, sec-butyl alcohol (2-butanol), in mg/100 ml of anhydrous alcohol and inorganic contaminants: Cu, Pb, As, Cd, and Mr. Mixed alcoholic beverages in the flavors of margherita and dulce de leche were also submitted to mandatory parameters to verify compliance with current norms of: real alcohol content, in %, in v/v, at 20 ºC, organic contaminants: methyl alcohol and inorganic contaminants: Cu and Pb. In addition to these parameters, the three

samples of the (standard) production batch were submitted to the parameters of total acidity, pH, total soluble solids (ºBrix), total sugars, ascorbic acid, metal analysis: Mg, k, Mn, Fe.

For the evaluation of shelf life, every 30 days the samples stored at 30 and 40 ºC up to a total period of 150 days were subjected to analysis of actual alcohol content, pH, total titratable acidity (TA), ascorbic acid, total sugars, total soluble solids (TSS), dry extract and instrumental color analysis.

3.2 Analysis for Mixed Drink (Cocktail) and Caipirinha

3.2.1 Alcoholic Degree

The determination of alcohol content was done through the refractometric method, this method was adapted from (AOAC, 2005). Refractometry is a physical method, where the refractive index of a solution varies regularly with the concentration of the solute, thus, the amount of ethanol in the solution was estimated through its refractive index (AOAC, method 950.04). The method used to determine the alcohol content involved a previous step of sample distillation, which was used a digital wine distiller model SUPER DEE, followed by the determination of ethanol by refractometry, the device used to read the alcohol content of the samples was from the brand e-LABShop, model RHW-80 with measurement from 0 to 80% v/v, with automatic temperature compensation (ATC) between 10 and 30 °C and accuracy of 1% ±. The refractometer was calibrated using distilled water, verifying that there was 0% alcohol in the reading, after which the samples were read directly, placing 2 drops in the prism, closing immediately so that there was no loss of components.

3.2.2 Total acidity

Total acidity is based on the neutralization reaction of acids with a standardized solution of alkali, up to the point of equivalence with the use of an indicator or potentiometer, up to pH = 8.2. For analysis, 25 mL of the sample was transferred to an Erlenmeyer flask containing 200 mL of distilled water, adding 2 to 3 drops of phenolphthalein. Then the sample was titrated with 0.1 N sodium hydroxide solution until pink. The total acidity result was expressed in grams of acetic acid per 100 mL of sample (g/100 mL). For caipirinha, the total acidity was expressed in grams of citric acid per 100 mL of sample (g/100 mL), (BRASIL, 2005b).

3.2.3 Inorganic contaminants

Inorganic contaminants were analyzed by plasma source mass spectrometry (ICP-MS) according to the determinations of (Adapted from Lawrence et al., (2006), the concentrations of the metals Mg, K, Mn, Fe, Cu, As, Cd, Sn and Pb, despite the fact that most of these contaminants are not part of the parameters of the legislation, there is a certain interest in their quantification, since the composition of beverages, especially cocktails, is very variable, allowing the characterization of different formulations. . With the aid of a plasma source mass spectrometer (ICP-MS), located at the Universidade Federal dos Vales do Jequitinhonha e Mucuri (UFVJM), the contaminants were analyzed under the following operating conditions: Nebulizer gas flows: 0.95 L/min; Auxiliary Gas Flow: 1.2 L/min; Plasma Gas flow: 15 L/min; Lens Voltage: 7.25V; ICP RF power: 1300 W; CeO/Ce = 0.011; Ba ++/Ba + = 0.016. To determine the concentrations of metals in the samples, the quantitative method was used, applying a multi-element calibration method containing: a standard at a concentration of 10 mg ml-1 for the nine metals (Matrix: 5% HNO3, Perkin Elmer), a standard of 10 mg ml-1 of Hg Matrix: 5% HNO3, Perkin Elmer) and a standard of 10 mg ml-1 of rare metals (Matrix 5% HNO3. The metals analyzed were: Mg, k, Mn, Fe, Cu, As, Cd, Sn and Pb. The samples were prepared by diluting 250 µL by 40 times in 2% nitric acid (v/v), then taken for quantification.

3.2.4 Inorganic contaminants

Inorganic contaminants were analyzed by plasma source mass spectrometry (ICP-MS) according to the determinations of (Adapted from LAWRENCE et al. 2006), the concentrations of the metals Mg, K, Mn, Fe, Cu, As, Cd, Sn and Pb, despite the fact that most of these contaminants are not part of the parameters of the legislation, there is a certain interest in their quantification, since the composition of beverages, especially cocktails, is very variable, allowing the characterization of different formulations.

With the aid of a plasma source mass spectrometer (ICP-MS), located at the Universidade Federal dos Vales do Jequitinhonha e Mucuri (UFVJM), the contaminants were analyzed under the following operating conditions: Nebulizer gas flows: 0.95 L/min; Auxiliary Gas Flow: 1.2 L/min; Plasma Gas flow: 15 L/min; Lens Voltage: 7.25V; ICP RF power: 1300 W; CeO/Ce = 0.011; Ba ++/Ba + = 0.016.

To determine the concentrations of metals in the samples, the quantitative method was used, applying a multi-element calibration method containing: a standard at a concentration of 10 mg ml-1 for the nine metals (Matrix: 5% HNO3, Perkin Elmer), a standard of 10 mg ml-1 of Hg Matrix: 5% HNO3, Perkin Elmer) and a standard of 10 mg ml-1 of rare metals (Matrix 5% HNO3. The metals analyzed were: Mg, k, Mn, Fe, Cu, As, Cd, Sn and Pb. The

samples were prepared by diluting 250 µL by 40 times in 2% nitric acid (v/v), then taken for quantification.

3.2.5 Methyl alcohol

The analysis of methanol in alcoholic beverages was performed using the spectrophotometric method, where methanol is oxidized by potassium permanganate to formaldehyde, which reacts with the chromotropic acid salt to give a spectrophotometrically measured purple color. The calculations were performed and expressed in mL of methyl alcohol per 100 mL of anhydrous alcohol, according to the formula (BRASIL, 1986).

3.2.6 pH

The pH was analyzed by the potentiometric method, according to (BRASIL, 2005b). A pH meter was used for the analysis, the temperature of which was between 20 and 25 °C and as close as possible to 20 °C. The electrode was immersed in the beaker containing the homogenized sample and the reading was performed at 20º C ± 2º C. The result was expressed as a pH value with two decimal places.

3.2.7 Total soluble solids (ºBrix)

The analysis of total soluble solids was performed by refractometric reading of the degrees °Brix of the sample at 20ºC (BRASIL, 2005b). To perform this analysis, we used an Abbe refractometer with a ºBrix degree scale with divisions of at least 0.2.

3.2.8 Total Sugars

For the analysis of total sugars, the titrimetric method (Eynon Lane method) was used. The non-reducing sugars were submitted to previous hydrolysis in an acid medium, dissociating the disaccharides into their monosaccharides, which react with the cupric ions of the Felling solution, reducing them to cuprous ions, under the action of heat in an alkaline medium. When reacting with cupric ions, the sugars undergo oxidation, while Cu(II) is reduced to Cu(I), forming a red precipitate of cuprous oxide, according to the methodology of (IAL, 2008).

3.2.9 Ascorbic acid

The determination of ascorbic acid was performed by the titrimetric method with potassium iodate. The method has its principle in the oxidation of ascorbic acid by titration with potassium iodate in a medium acidified with sulfuric acid in the presence of potassium iodide and starch solution, the analysis was determined according to the (IAL, 2008).

3.2.10 Color analysis

The color test was carried out in the physical-chemical analysis laboratory of the Federal Institute of Science and Technology of the North of Minas – Campus Salinas. After the sensory test, the beverage samples were stored at a temperature around 5 °C in order to avoid changes in their color.

Color analysis was performed according to RAVINDRANATH et al., 2018, with some adaptations. A Samsung Galaxy A31 smartphone was used with a resolution of 2400x1080 pixel, 48-megapixel camera that allows a resolution of 8000x6000 pixels, in this device the colorimeter application (Lab tools version 1.6.6.4) was installed, available in the application store. for Android OS smartphones (*PLAY STORE*, 2022).

The values of L*, a*e b*, C* and h given by the smartphone were measured and the color space adopted for the interpretation of the results was the CIELAB. In the CIELAB color system, L* represents luminosity, where values range from 0 (black) to 100 (white). Also, -a* (green), a* (red), -b* (blue), and b* (yellow) are the color coordinates. The parameters C* and h are derived from the previously mentioned colorimetric coordinates, where C* is the chromaticity or color saturation ("brightness") and h indicates the color tone, whose measurement is given in degrees.

The center of the CIELAB color space is achromatic and the color saturation increases as the values move away from the origin (Rossini et al., 2012). From each sample, 4 replicates were collected, with 6 samples referring to each storage time for a period of 150 days divided into 5 times plus the standard sample. A light box was created to standardize the light intensity directed towards the sample at the time of data collection, this light falls on the four sides of the box generating about 2100 lux measured in a SMART SENSOR brand luxmeter model: AS803, the device has a measurement range 0-200,000 Lux, resolution of 1 lux, sampling rate of 1.5 times/s and measurement repeatability of about 2%. In order to avoid interference from the surface where the 15 cm diameter Petri dish was placed, a mirror was placed under the sample, with the intention of obtaining a color without external interference.

For the actual color analysis, the sample was homogenized and 40 mL was measured in each Petri dish, positioning it in the center of the rectangular box that has an area for the smartphone made for this purpose. The collection shots were performed at about 20 cm from the sample in all readings, after which the data were exported in an Excel spreadsheet to the computer to receive the treatments.

3.3 Reviews for Caipirinha

For the determination of congener coefficient parameters (expressed in mg/100ml of anhydrous alcohol), aldehydes, in acetaldehyde, in mg/100 ml of anhydrous

alcohol, total esters in ethyl acetate, ethyl lactate, higher alcohols (expressed by sum of n-propyl alcohol, isobutyl alcohol and isoamyl alcohol), (furfural + 5-hydroxymethylfurfural); acrolein (2-propenal), in mg/100 ml of anhydrous alcohol; n-butyl alcohol (1-butanol), in mg/100 ml of anhydrous alcohol; sec-butyl alcohol (2-butanol), in mg/100 ml of anhydrous alcohol and ethyl carbamate, in µg/l, parameters charged by Decree 6,871/2009, art. 68, § 5, IN MAPA 35/2010, arts. 8 and 17 to 21, IN MAPA 13/2005 and Resolution RDC 42/2013, the caipirinha pattern sample was sent to the Laboratory Amazile Biagioni Maia Ltda (LABM) in Belo Horizonte MG), according to the description of the analytical methods of the analysis of the sample caipirinha ref. LABM 1569/2. The results of the analyses, Annex A, were sent by the company, carried out in duplicate and do not have standard deviation.

The coefficient of congeners (expressed in mg/100ml of anhydrous alcohol), aldehydes, total esters, ethyl lactate, higher alcohols (n-propyl alcohol, isobutyl alcohol and isoamyl alcohol), (furfural + 5-hydroxymethylfurfural); acrolein; n-butyl alcohol, sec-butyl alcohol were analyzed by gas chromatography with flame ionization detector (GC-FID). For the analysis, a GCROM Generation 8000 chromatograph equipped with a flame ionization detector (CG-FID) is used. The chromatography column used is DB-WAXETR from AGILENT. The oven temperature setting is 3 minutes at 35 °C (3.0 min), 35-80 °C (5.0 °C/min), 80 °C (3 min), 80-160 °C (6 .1°C/min). The inlet and detector temperatures are 140 °C and 180 °C, respectively. The injection volume is 2 µL in split mode (1:1) and the carrier gas is nitrogen (6.0 mL/min).

Ethyl carbamate was quantified by gas chromatography coupled to a mass spectrometer. An AGILENT GC 4350A gas chromatograph was used, equipped with a 7036A mass detector and a G4513A automatic injector, operating in electronic impact mode at 70eV, with monitoring of the selective ion m/z 62. The HP- AGILENT's FFAP. The carrier gas helium at a flow of 1.5mL/min; Temperature programming starts at 90°C (2min) high (14 minutes - 10.7°C/min) up to 240°C (10 min). The inlet temperature is 230 °C and the detector temperature is also 230 °C. The injection volume was 2 µL (direct injection of the sample distillate), the results are shown in Annex B.

IV. STATISTICAL ANALYSIS

Physicochemical analyzes were performed in triplicate. The results obtained were submitted to the Restudio statistical program and the Shapiro-Wilk normality tests were applied, the descriptive statistics in the Tukey test and the non-parametric data, the Kruskal Wallis test, both at a 5% significance level. The color analysis data were submitted to the Shapiro Wilk normality test, the ANOVA was performed by Kruskal Willis where the data were identified as non-parametric and the analysis of variance by the Tukey test, both at a 5% significance level in the program Minitab version 19.1, LLC All rights reserved.

V. RESULTS AND DISCUSSIONS

1.1 Evaluation of the formulation of bottled beverages in relation to physical-chemical aspects

Physicochemical analyzes were carried out on the drinks after their manufacture, the caipirinha being called C-T0, the margherita M-T0 and the dulce de leche DL-T0, the results are organized in Tables 1, 2 and 3, respectively, as well as their comparisons with the official parameters and their maximum and minimum standards governed by the legislation of the Ministry of Agriculture, Livestock and Supply (BRASIL, 2009) through the provisions of Decree No. 6,871, of June 4.

Table 1: Official physical-chemical parameters versus laboratory analysis of the caipirinha sample

parameters	**Standards**		**Result of analysis**	**detectable limit**
	Minimum	Maximum		
Alcohol content in v/v, at 20ºC	≥ 15	≤ 36	17,9 ± 0,05	0,5
Titratable acidity in g/100 g	1	---	0,51 ± *	---
Sugar, in sucrose, in g/L	≥ 10	≤ 150	136,44	---
Congener coefficient, mg/100 mL aa	200	650	229,6	---
sweeteners	Absent		ND	---
organic contaminants	**Minimum**	**Maximum**		
Methyl alcohol, in mL/100 mL of aa	---	20	0,065 ± 0,6	---
Ethyl carbamate, in µg/L	---	210	< LQ	50
Acrolein (2-propenal), in mg/100 mL of anhydrous	---	5	< LQ	1,5
Sec-butyl alcohol (2-butanol), in mg/100 mL of	---	10	< LQ	1,7
n-Butyl alcohol (1-butanol), in mg/100 mL of	---	3	1,6	0,9

inorganic contaminants	**Minimum**	**Maximum**		
Copper, in mg/L	---	5	0,055 ± 0,2	---
Lead, in µg/L	---	200	1,70 ± 0,25	---
Arsenic, in µg/L	---	100	≤LD	---
Cadmium, in mg/kg	---	0,02	≤LD	---

Source: Adapted from(BRAZIL, 2009)

*: values that do not meet the legislation; LQ: Limit of Quantification; ---: no maximum or minimum limits; ≤LD: less than or equal to the detectable limit; mg/100 mL ethanol: milligram per 100 milliliters of ethanol; % v/v at 20 °C: percentage volume by volume at 20 °C; µg/L: microgram per litre.

Table 2: Official physicochemical parameters versus laboratory analysis of the margherita sample

parameters	**Standards**		**Result of analysis**
	Minimum	**Maximum**	
Alcohol content, de a.a.	≥ 0,5	≤ 54	15,95 ± 0,05
sweeteners	Absent		ND
contaminants	**Minimum**	**Maximum**	
Methyl alcohol, mg/100 mL de a.a.	---	200	0,03 ± 0,008
Copper, in mg/L	---	5	0,0693±1,639
Lead, in mg/L	---	0,2	≤LD

Source: Adapted from(BRAZIL, 2009).

---: no maximum or minimum limits; ≤LD: less than or equal to the detectable limit; mg/100 mL ethanol: milligram per 100 milliliters of ethanol; % v/v at 20 °C: percentage volume by volume at 20 °C; ND: not determined; a.a: anhydrous alcohol.

Table 2: Official physicochemical parameters versus laboratory analysis of the dulce de milk sample.

Parameters	**Standards**		**Result of analysis**
	Minimum	**Maximum**	
Alcohol content, in %, in v/v, at 20 °C	≥ 0,5	≤ 54	15,00 ± 0,05
sweeteners	Absent		ND
Contaminants	**Minimum**	**Maximum**	
Methyl alcohol, in mg/100 mL of anhydrous alcohol	---	200	0,13 ± 0,001
Copper, in mg/L	---	5	0,0868±14,68
Lead, in mg/L	---	0,2	0,0021± ,270

Source: Adapted from(BRAZIL, 2009)

---: no maximum or minimum limits; mg/100 mL ethanol: milligram per 100 milliliters of ethanol; % v/v at 20 °C: percentage volume by volume at 20 °C; ND: not determined.

In the comparisons carried out (Table 1), it is shown that the caipirinha meets the quality identity parameters of the Ministry of Agriculture, Livestock and Food Supply, except for the acidity parameter titratable in citric acid, in which the legislation allows at least 1 g/ 100 mL of sample and 0.518 g/100 mL of sample was found. According to Normative Instruction No. 35 (BRASIL, 2010) beverages must meet the identity and quality standards defined in current legislation, however, the legislator does not define in this Normative Instruction a punishment for non-compliance with this requirement. The drink will be adapted to identity and quality standards by adding a certain amount of lemon pulp to meet the minimum limit of citric acid in the titratable acidity.

As for the analysis of "sweeteners", despite being a mandatory parameter, both in caipirinha and in mixed

alcoholic beverages, it was not carried out, given that no type of sweetener is added in the formulation of the drinks, the result would certainly be absent. The samples of margherita and dulce de milk, Tables 2 and 3, respectively, freshly prepared, fit the quality identity standards in the Ministry of Agriculture, Livestock and Supply.

Table 4 shows the content of a series of chemical elements in the three samples of alcoholic beverages. Only Cu, Pb, Ar and Cd have maximum and minimum limits required by current legislation. Although most of these chemical elements are not considered as contaminants by the current legislation, there was a need to know their contents based on the proposed formulation.

Table 3: Concentrations of chemical elements analyzed via ICP-MS in standard samples.

Chemical element		C-T0	M-T0	DL-T0
arsenic	mg/L	≤LD	0,0025 ± 0,0004	0,0058 ± 0,008
Cadmium	mg/L	≤LD	≤LD	≤LD
Lead	mg/L	0,0017 ± 0,003	≤LD	0,0021 ± 0,003
Copper	mg/L	0,0554 ± 0,0063	0,0694 ± 0,0016	0,0902 ± 0,014
Tin	mg/L	≤LD	≤LD	≤LD
Iron	mg/L	0,17 ± 0,030	0,25 ± 0,25	0,31 ± 0,044
Magnesium	mg/L	15,87 ± 1,51	18,59 ± 0,40	23,75 ± 3,42
Manganese	mg/L	0,10 ± 9,64	0,14 ± 7,36	0,29 ± 91,94
Potassium	mg/L	223,38 ± 21,42	327,90 ± 23,00	574,99 ± 0,159

≤LD: less than or equal to the detectable limit; mg/L of sample: milligram per liter of sample.

The results found for the contaminants Cu, Pb, Ar and Cd of the 3 alcoholic beverages are well below the minimum limits established by the Ministry of Agriculture, Livestock and Supply. Because the minimum concentrations found are considered as contaminants, there is no implication, on the contrary, their absence characterizes the quality of the beverage.

According to (Lima & Filho, 2011), the presence of some chemical compounds makes it difficult to prepare beverages, so some of these elements must be controlled, such as manganese (0.3 mg/L-1), lead (0.1 mg /L-1) and iron (0.1 mg/L-1). In the three formulations, a higher iron content was found than recommended by (Lima & Filho, 2011), which can cause precipitation of salts, reaction with dyes and aroma/flavor substances.

1.2 Color analysis

Instrumental color analysis is usually performed in colorimeter-type devices, however, smartphones are currently being used as an alternative tool for colorimetry, as described in the literature by several authors, such as: (Almeida, 2021; Cerutti; et al., 2018; Cunha, 2019; Lucas et al., 2021; Ravindranath et al., 2018). This technology was used in this work in order to analyze the variation of color parameters between the storage times of the samples at the two temperatures evaluated. The color parameters L, Chroma and HUE were analyzed to elucidate the reactions that occurred in samples of caipirinha margherita and dulce de milk stored at 30 and 40 °C for a period of time from zero to 150 days.

Figure 1 represents the way of reading the dispositions in the CIELAB color system, where L* represents the luminosity, its values vary from 0 (black) to 100 (white), -a* (green), a* (red), -b* (blue) and b* (yellow) these last two are the color coordinates. The parameters C* and h are derived from the previously mentioned colorimetric coordinates, C* is the chromaticity or saturation of the color ("brightness") and h (HUE) indicates the tone of the color, whose measurement is given in degrees, (ROSSINI et al., 2012).

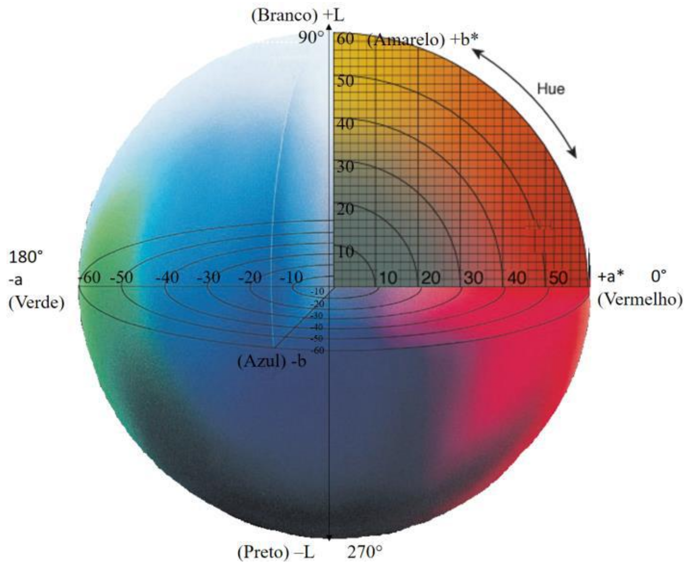

Fig.1: Representation of the CIE L, a*, b*, chroma and HUE color space.*

Source: Adapted from (Minolta, 1990)

Tables 4 and 5 show the comparison of means and standard deviation of the results of the color analysis for caipirinha, margherita and dulce de leche stored at 30 °C and 40 °C, respectively.

Table 4. Means, standard deviation and comparison of means by the Tukey/Kruskal Willes test at the 5% significance level of samples of caipirinha, margherita and dulce de milk stored at 30 °C.

Time		CAIPIRINHA			MARGHERITA			DULCE DE LEITE		
		L	CROMA	HUE	L	CROMA	HUE	L	CROMA	HUE
averages Standard deviation	T0	**60,13 ^{a} ± 0,54**	16,04 ^{c} ±	149,40^{b}±0,5	**52,95 ^{e} ± 0,25**	26,99^{c} ± 0,55	103,47 ^{a} ±	**44,20 ^{c}±0,61**	34,54	42,55 ^{a} ± 0,90
	T1	**60,63 ^{a} ± 0,96**	15,52 ^{c} ±	153,55^{a}± 2,0	**60,57 ^{a} ±1,86**	27,32 ^{c} ±0,17	62,40 ^{c} ± 0,00	**55,40 ^{a}±0,95**	40,20^{c}	37,15 ^{b}± 0,38
	T2	**58,98 ^{b} ± 0,38**	19,05 ^{b} ±	138,47 ^{c}	**59,00 ^{b} ± 0,5**	27,68 ^{c} ± 0,3	63,60 ^{c} ± 1,39	**53,88 ^{b} ± 0,6**	44,26^{b}	36,42 ^{b} ±1,21
	T3	**59,40 ^{ab}±0,41**	18,24 ^{b} ±	141,00^{c} ±1,8	**62,80 ^{a} ±0,36**	32,36 ^{a} ±1,32	93,50 ^{b} ± 1,01	**55,89 ^{a} ±0,6**	46,54	36,80 ^{b} ±0,71
	T4	**59,75 ^{ab}±1,97**	21,40 ^{a} ±	122,40 ^{d}	**56,20 ^{d} ±0,1**	30,01 ^{b} ±0,60	63,37 ^{c} ± 0,06	**55,93 ^{a} ±0,0**	37,34^{d}	43,73 ^{a} ±0,68
	T5	**59,05 ^{b} ±0,68**	21,45 ^{a} ±	120,00 ^{d}	**58,33 ^{c} ±0,39**	30,67 ^{b} ±0,65	61,92 ^{c} ± 1,65	**55,93 ^{a} ±0,0**	37,94^{d}	35,90^{b} ± 0,00

Means that do not share the same letter per column are statistically different at the 5% significance level; data in bold indicate nonparametric distribution and were performed in Kruskal Willis.

Table 5. Means, standard deviation and comparison of means by the Tukey/Kruskal Willis test at the 5% significance level of samples of caipirinha, margherita and dulce de leche stored at 40 °C.

Time	CAIPIRINHA			MARGHERITA			DULCE DE LEITE		
	L	CROMA	HUE	L	CROMA	HUE	L	CROMA	HUE
Standard deviation	60.1 b ±0.538	16.4 c ± 0.82	149 a ±	53.0 d ±0.2	27.0 f ± 0.54	103 a ±	44.2 a ± 0.61	34.5 d ± 0.63	42.5a ± 0.90
	65,1 a ±0,465	16,7 c ± 0,59	140 b ±	56,0 c	32,0 e ± 0,37	59,7 b ±1,1	37,6 c ± 2,22	39,6 bc ±	33,4c ± 0,85
	64,3 a ±0,545	24,7 b ± 0,63	102 c	58,5 b ±0,8	35,0 d ± 0,55	60,0 b ±0,0	41,2 bc ± 0,18	41,4 ab ±	33,4c ± 0,58
	59,1 c ±0,189	31,4 a ± 0,26	59,0 e	56,4 c	43,4 a ± 0,95	56,1 c	41,2 bc ± 0,77	42,1 a ± 0,52	31,7c ± 0,73
	55,9 d ±0,189	25,2 b ± 0,82	61,7 d	59,8 a ±0,4	37,6 c ± 0,75	58,9 b ±1,2	35,5 c ± 5,59	37,3 c ± 3,14	35,5b ± 1,46
	60,8 b ±0,271	24,0 b ± 0,70	63,5 d	61,3 a ±2,5	41,6 b ± 0,18	58,3 bc	42,8 ab ± 0,37	31,5 d ± 0,11	41,6a ± 0,30

Means that do not share a letter per column are statistically different at the 5% significance level; data in bold indicate nonparametric distribution and were performed at Kruskal Willis

Through the values described in Tables 4 and 5, the luminosity parameter (L) of the caipirinha samples showed a significant difference over the storage time, in the same way the chroma and the HUE showed changes in color at the level of 5 % of significance. (Harder et al., 2007) state that the chroma values can be interpreted as the ratio of the parameters a* and b*, that is, the real color of the samples, its reading is performed from the center (zero) to the ends; consequently, the caipirinha samples showed a significant change in color, becoming more saturated. The value of the HUE angle of the caipirinha reduced significantly over time, following the green hue, with the passage of time it became more yellow.

The margherite samples stored at 30 °C showed an oscillatory luminosity behavior, possibly caused by reactions that were still in progress, but it can be observed, in general, that this parameter increased significantly over time, in most of the analyzed times, as seen in Table 5. These oscillations were also noticed in the chroma and HUE of the same sample, these results being significant in the color variation of the samples. Martins (2009) found a similar oscillation behavior, however in the total color difference. The chroma increased significantly at the 5% level of significance, indicating an increase in the saturation of the sample, and the Hue angle also reduced significantly, at first the sample was yellowish, becoming orange over time.

The color changes suffered by the dulce de leche samples shown in Table 4, express well the results obtained in the analysis of comparison of color averages, where the luminosity showed a significant change in T0 in relation to the other 5 storage times at 30 °C and chroma showed significant increases, no similarity was seen in any of the dulce de leche samples stored at 30 °C, the HUE value underwent significant reductions by Tukey's test at 5% of significance.

Storage time and temperature act directly on the darkening of the analyzed samples, this is noticed when Tables 4 and 5 are compared. For caipirinha, the L values, despite fluctuating, were similar at the beginning and end of storage. Chroma increased and HUE values decreased, at the 5% significance level, indicating saturation and color change (from greenish to orange) during storage. The behavior of margherita samples stored at 40 °C was similar to that of caipirinha samples at 30 °C, with regard to chroma and HUE parameters. Brightness and saturation increased, while the hue went from yellowish to orange.

The behavior of margherita samples stored at 40 °C was similar to that of caipirinha samples at 30 °C, with regard to chroma and HUE parameters. Brightness and saturation increased, while the hue went from yellowish to orange.

The dulce de leche samples stored at 40 °C (Table 5) were presented in an unusual way, the results obtained variations at the level of 5% significantly over the storage time in the 3 parameters of the color space analyzed L, chroma and HUE. However, in general, it can be seen that the samples, close to 150 days of storage, had a tendency to initial color.

REFERENCES

[1] ALMEIDA, G. A. DE. (2021). *METODOLOGIA DE BAIXO CUSTO COM USO DO SMARTPHONE NO MONITORAMENTO DA CONCENTRAÇÃO DE NITRITO EM EMBUTIDOS CÁRNEOS*. ANVISA. (2010). *Resolução ANVISA nº 24/2010. Dispõe sobre os critérios para a divulgação de produtos alimentícios.* 1–7. http://portal.anvisa.gov.br/wps/wcm/connect/34565380474597549fd4df3fbc4c6735/RDC24_10_Publicidade+de+alimentos.pdf?MOD=AJPERES

[2] BRASIL, Ministério da Agricultura, P. e A. – M. S. de D. A. – S. C. G. de A. L.-C. (1986). *Manual de Métodos de Análises de Bebidas e Vinagres*. 1986.

[3] Resolução da Diretoria Colegiada – RDC nº 2, de 15 de janeiro de 2007, 9 10 (2007). https://www.infodesign.org.br/infodesign/article/view/355%0Ahttp://www.abergo.org.br/revista/index.php/ae/article/view/731%0Ahttp://www.abergo.org.br/revista/index.php/ae/article/view/269%0Ahttp://www.abergo.org.br/revista/index.php/ae/article/view/106

[4] Brasil. (2005). *Manual de Métodos de Análises de Bebidas e Vinagres*. https://www.gov.br/agricultura/pt-br/assuntos/laboratorios/legislacoes-e-metodos/arquivos-metodos-da-area-bev-iqa/ferm-alc-04-ph.pdf

[5] Brasil. (2013). *Ministério da Saúde. Agência Nacional de Vigilância Sanitária. 2013. Resolução-RDC, número 5. Aprova o uso de aditivos alimentares com suas respectivas funções e limites máximos para bebidas alcoólicas (exceto as fermentadas). Diário Oficial da União, Pod. 12*, Seção 1, 43.

[6] INSTRUÇÃO NORMATIVA Nº 13, DE 29 DE JUNHO DE 2005 Aprova o Regulamento Técnico para Fixação dos Padrões de Identidade e Qualidade para Aguardente de Cana e para Cachaça, 5 (2005).

[7] BRASIL. (2009). *Decreto N°6.871 de 2009 - Regulamenta a Lei n° 8.918, de 14 de julho de 1994, que dispõe sobre a padronização, a classificação, o resgistro, a inspeção, a produção e a fiscalização de bebidas.* https://cutt.ly/Mht31vQ

[8] BRASIL. (2010a). Manual de Métodos de Análises de Bebidas e Vinagres, caderno 4 Fermentados Alcoólicos. *Ministério da Agricultura, Pecuária e Abastecimento-MAPA. Secretaria de Defesa Agropecuária- SDA, Coordenação Geral de Apoio Laboratorial-CGAL, d*, 1–2.

[9] Consolidação das Normas de Bebidas, Fermentado Acético,

Vinho e Derivados da Uva e do Vinho., 10 Ministério da Agricultura Pecuária e Abastecimento 813 (2019). https://cutt.ly/2ht1qUE

[10] BRASIL, M. da A. P. e abastecimento. (2010b). INSTRUÇÃO NORMATIVA Nº 35, DE 16 DE NOVEMBRO DE 2010. *MINISTÉRIO DA AGRICULTURA, PECUÁRIA E ABASTECIMENTO*, *9*(1), 76–99.

[11] Cerutti;, P. H., Wille;, C. L., Adams;, C. R., Barichello;, E. C., Santos;, M. dos, & Gemeli., M. S. (2018). Uso de smartphones como ferramenta de baixo custo na avaliação de cor em frutos. *Revista da 15ª Jornada de Pós graduação e Pesquisa*, *15*, 1543–1555. http://revista.urcamp.tche.br/index.php/rcjpgp/article/view/2927

[12] Cunha, T. J. N. (2019). *Smartphone como sensor colorimétrico de baixo custo* [Universidade de Lisboa]. https://fenix.tecnico.ulisboa.pt/cursos/meec/dissertacao/1409728525632841

[13] FILHO, O. G. (2003). AVALIAÇÃO DA PRODUÇÃO ARTESANAL DA AGUARDENTE DE BANANA UTILIZANDO Saccharomyces cerevisiae CA-1174. Tese (Doutorado em Ciência dos Alimentos). In *Universidade Federal de Lavras-MG*.

[14] FONSÊCA, L. D. S. M. DA. (2020). *ESTUDO PROSPECTIVO E EXPLORATÓRIO SOBRE AS CACHAÇAS DE ALAMBIQUE DE ALAGOAS E SUAS POTENCIALIDADES PARA A INDICAÇÃO GEOGRÁFICA E SEUS DIFERENCIAIS COMPETITIVOS* [Universidade Federal de Alagoas]. http://www.repositorio.ufal.br/bitstream/riufal/7222/3/Estudo prospectivo e exploratório sobre as cachaças de alambique de Alagoas e suas potencialidades para a Indicação geográfica e seus diferenciais competitivos.pdf

[15] Giménez, A., Ares, F., & Ares, G. (2012). Sensory shelf-life estimation: A review of current methodological approaches. *Food Research International*, *49*(1), 311–325. https://doi.org/10.1016/j.foodres.2012.07.008

[16] Gonzaga, A. M., & Moreira, M. M. da S. (2008). *CONCEITOS CIENTÍFICOS E PROPAGANDAS DE TV NAS SÉRIES*. 73–87.

[17] Harder, M. N. C., Canniatti-Brazaca, S. G., & Arthur, V. (2007). Avaliação quantitativa por colorímetro digital da cor do ovo de galinhas poedeiras alimentadas com urucum (Bixa orellana). *Rpcv*, *102*(563–564), 339–342. http://www.fmv.ulisboa.pt/spcv/PDF/pdf12_2007/339-342.pdf

[18] Horwitz, W., & George W. Latimer, J. (2005). Official Methods of Analysis of AOAC INTERNATIONAL. *Aoac*, *February*.

[19] INSTITUTO ADOLFO LUTZ. Normas Analíticas do Instituto Adolfo Lutz. Métodos físico-químicos para análises de alimentos. 4ª ed. (1ª Edição digital), 1020 p., (2008). https://cutt.ly/RjDeKnd

[20] Lawrence, M. G., Greig, A., Collerson, K. D., & Kamber, B. S. (2006). Direct quantification of rare earth element concentrations in natural waters by ICP-MS. *Applied Geochemistry*, *21*(5), 839–848. https://doi.org/10.1016/j.apgeochem.2006.02.013

[21] Lima, L. L. de A., & Filho, A. B. de M. (2011). Tecnologia de bebidas. In *e-Tec Brasil*. https://www.google.com.br/url?sa=t&rct=j&q=&esrc=s&source=web&cd=1&cad=rja&uact=8&ved=0ahUKEwiZ1Kii1YvQAhUMlZAKHbprDekQFggdMAA&url=%2Finterstitial%3Furl%3Dhttp%3A%2F%2F200.17.98.44%2Fpronatec%2Fwp-content%2Fuploads%2F2013%2F06%2FTecnologia_de_Bebidas.pdf&

[22] Lucas, B. N., Schú, A. I., & Dalla Nora, F. M. (2021). Uso De Smartphone Como Alternativa Inovadora No Controle De Qualidade De Alimentos: Uma Breve Revisão. *Avanços em Ciência e Tecnologia de Alimentos - Volume 3*, *3*, 278–288. https://doi.org/10.37885/210203310

[23] MARTINS, G. A. D. S. (2009). DETERMINAÇÃO DA VIDA-DE-PRATELEIRA POR TESTES ACELERADOS DE DOCE EM MASSA DE BANANA CV. PRATA. dissertação (Mestrado em em Ciência dos Alimentos). Lavras-MG. In *Universidade Federal de Lavras-MG*. Universidade Federal de Lavras.

[24] Minolta, K. (1990). Historical development of CIE recommended color difference equations. *Color Research & Application*, *15*(3), 167–170. https://doi.org/10.1002/col.5080150308

[25] MIRANDA. (2007). Chemical quality of brazilian sugarcane spirits. *Ciência e Tecnologia de Alimentos, 27(4): 897-901*, *27*(4), 897–901.

[26] Naconha, A. E. (2021). *Novas linhagens de Saccharomyces cerevisiae e híbridos entre S. cerevisiae e Saccharomyces kudriavzevii para a produção de cerveja, cachaça e vinho*. *4*(1), 6.

[27] *PLAY STORE Google Android Play Store . Disponível em: https://play.google.com/store>. 2022*.

[28] Ravindranath, R., Periasamy, A. P., Roy, P., Chen, Y. W., & Chang, H. T. (2018). Smart app-based on-field colorimetric quantification of mercury via analyte-induced enhancement of the photocatalytic activity of TiO2–Au nanospheres. *Analytical and Bioanalytical Chemistry*, *410*(18), 4555–4564. https://doi.org/10.1007/s00216-018-1114-7

[29] Rossini, K., Anzanello, M. J., & Fogliatto, F. S. (2012). Seleção de atributos em avaliações sensoriais descritivas. *Production*, *22*(3), 380–390. https://doi.org/10.1590/s0103-65132012005000032

[30] Schneider, D., Rasch, D., Deisi, ;, Dewes, C., Érica De Souza, ;, Korbes, J., Luciane, ;, Hammes, D., & Piletti, R. (2018). *DETERMINAÇÃO DE VIDA-DE-PRATELEIRA DE PRODUTOS ALIMENTÍCIOS*. 6.

[31] VIANA, E. J. (2016). DIAGNÓSTICO DA CADEIA PRODUTIVA E AVALIAÇÃO FÍSICO-QUÍMICA DE CACHAÇAS DO ESTADO DA BAHIA. In *UNIVERSIDADE ESTADUAL DO SUDOESTE DA BAHIA – Uesb Programa De Pós-Graduação Em Engenharia E Ciência De Alimentos*.